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Surface Modification of Steel Using Automotive Waste as Raw Materials

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Abstract
It is usually not economically feasible to utilize conventional sorting and recycling techniques to treat complex industrial waste due to the heterogeneous nature thereof and hence, it is mostly discarded of by landfilling. In the present study, complex wastes derived from the automobile industry have been used as an input stream for the modification of steel surfaces. High abrasion and corrosion resistance steels have attracted much interest in industrial applications because costs associated with product failure can be minimized. Although these classes of steel increase the abrasion and corrosion resistance, they contain expensive alloying elements that increase cost and in addition, complicate production techniques. For this reason, several surface hardening techniques have been developed to improve the abrasion and corrosion resistance of less expensive carbon steel, but these techniques are in general not cost-effective. In this study, a complex industrial waste stream has been transformed in situ via precisely controlled high temperature reactions to produce a chemically-bonded ceramic surface on normal carbon steel. The ceramic surface increases both the compressive strength of the steel, and therefore its abrasion and corrosion resistance. Furthermore, by modifying the composition of the waste input and the processing parameters, the ceramic surface can be effectively customized to match the intended application of the steel. This economical new approach marries industry demands for more cost-effective, durable steel products with global imperatives to address resource depletion and environmental degradation through the recovery of resources from waste.

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Surface Modification of Steel Using Automotive Waste as Raw Materials

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Abstract

It is usually not economically feasible to utilize conventional sorting and recycling techniques to treat complex industrial waste due to the heterogeneous nature thereof and hence, it is mostly discarded of by landfilling. In the present study, complex wastes derived from the automobile industry have been used as an input stream for the modification of steel surfaces. High abrasion and corrosion resistance steels have attracted much interest in industrial applications because costs associated with product failure can be minimized. Although these classes of steel increase the abrasion and corrosion resistance, they contain expensive alloying elements that increase cost and in addition, complicate production techniques. For this reason, several surface hardening techniques have been developed to improve the abrasion and corrosion resistance of less expensive carbon steel, but these techniques are in general not cost-effective. In this study, a complex industrial waste stream has been transformed in situ via precisely controlled high temperature reactions to produce a chemically-bonded ceramic surface on normal carbon steel. The ceramic surface increases both the compressive strength of the steel, and therefore its abrasion and corrosion resistance. Furthermore, by modifying the composition of the waste input and the processing parameters, the ceramic surface can be effectively customized to match the intended application of the steel. This economical new approach marries industry demands for more cost-effective, durable steel products with global imperatives to address resource depletion and environmental degradation through the recovery of resources from waste.

Peer-review under responsibility of the organizing committee of SMPM 2017

Keywords: Ceramic surface; Surface modification of steel; Automotive waste;

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1. Introduction

The present study is primarily aimed at utilizing some of the materials present in automotive shredder residue (ASR) in order to produce steel components with an increased wear and corrosion resistance. In this respect the study is economically and environmentally significant since it purports to usefully employ automotive waste as a means of providing a cost-effective technique for improving the surface hardness of steel components whilst at the same time, reduces the amount of waste going to landfill.

Automotive Shredder Residue (ASR) generally consists of materials such as plastic, rubber, wood, fabric, non-ferrous metals, leather, glass, paper, textiles and dirt [1], all of which have the potential of being recovered. In the present project plastic waste derived from the automobile industry will be utilized as a source of carburizing and/or nitriding gasses that can be used to modify the surface of steel components. The waste input is sourced from end-of-life vehicles. Every year tens of millions of vehicles are decommissioned from the 1.4 billion or so vehicles on the world's roads. Reusable parts are removed manually for recycling or other uses, depending upon their age and value. Then, ferrous and non-ferrous metals are separated by magnetic separation and eddy current separation respectively. Although the ferrous and other metals, which make up about 75% of the mass of a vehicle, can be readily and profitably, recovered and recycled, the remaining waste is mainly destined for landfill as Automotive Shredder Residue (ASR) [2]. For every car, some 100 – 200 kg of complex and potentially toxic shredded residue ends up in landfill, posing a growing environmental challenge worldwide, and representing a significant waste of finite resources. Moreover, landfills pose complex and serious problems, they have an adverse effect on surrounding land, and its value, since leaching of dangerous chemicals can threaten the surrounding environment and the health of local communities [3]. Dumping also disregards the potential of recovering scarce elements.

Ferrous metal components are susceptible to rapid wear and corrosion, which raise significantly the costs of scheduled and unplanned maintenance in industry and severe losses of functionality in a wide range of applications [4–6]. By increasing the wear and corrosion resistance of such metals their life can be extended and therefore replacement and maintenance costs can be minimized [7]. Several methods have been developed to improve the wear and corrosion resistance, but are not always cost effective. Two main approaches have been pursued: enhancing the resistance of the metal in bulk solid form by microstructural modifications or by employing surface engineering principles such as the application of coatings, films or surface modifications. The wear- and corrosion-resistance of ferrous metals can be improved by changes in microstructure [8, 9], by low-temperature carburizing [10], or by introducing a duplex microstructure through suitable heat treatments [11]. These processes are effective but there remain an urgent need to further improve wear and corrosion resistance without affecting bulk properties. One such technique involves optimization of the morphology and quantity of carbides on the surface by the addition of selected alloying elements [12 – 15].

Producing a highly-alloyed coating on a ferrous metal substrate is another effective measure [16, 17]. Common fabrication methods used to apply such coatings are composite casting [18], welding deposits [19] or cladding produce by hot-isostatic pressing techniques (HIP) [17, 20]. Composite casting and welding deposits are easy to apply, the processes are fast and cheap, but produce inhomogeneous hard-phase distributions compared to coatings applied by HIP cladding techniques [17, 20]. However, HIP processing techniques are expensive and an inflexible a technology and hence, the proposed use of automobile waste as a source of carburizing and/or nitrodizing gas by which steel surfaces can be hardened is an interesting prospect worth pursuing.

2. Experimental Procedure

In order to investigate the formation of a wear and corrosion resistant ('ceramic') surface on a steel component, a 0.4% C steel specimen 4mm in diameter and 10mm in length was used. The reactive gas used to surface-harden the steel specimen was derived from automotive shredder residue (ASR) with an average size of 2mm and a chemical composition shown in Table 1. The steel samples were put into a zirconia crucible, then filled and covered with raw ASR granules until the crucible was packed tightly. The crucible was then covered with a lid (of the same material as the crucible itself) in order to provide a closed reaction chamber. The crucible was then placed on a graphite rod and fed into a horizontal tube furnace. High purity argon gas was passed through the furnace at a rate of 1litre/min to

produce the required inert conditions. The crucible was then positioned in the cold-zone of the furnace at a temperature of about 250–300°C and kept there for about 10 minutes to prevent thermal shock. It was then transferred to the hot-zone in which a temperature of 1200°C was maintained. Following a soaking period of 30 and 60 minutes, the samples were transferred back into the cold-zone and kept for a further 15 minutes to prevent thermal cracking and the oxidation of the steel specimens.

Table 1: Chemical composition of automotive shredder residue

Elements	C	N	Ti	Si	Al
Raw ASR	19.43	0.72	2.68	0.49	0.1
ASR at 1200ºC	61.45	1.4	12.55	5.45	0.45

The heat-treated steel samples were then cut using a diamond cutter (Struers Minitom) and prepared for microstructural investigation using the standard metallographic sample-preparation techniques. The surface structure was analyzed by using X-ray photo-electron spectroscopy (XPS) in an ESCALAB 250Xi (a high-intensity XPS instrument which uses mono-chromated Al radiation (Ka hv = 1486.7 eV). Orientation microscopical investigations were conducted by using electron back-scattered diffraction (EBSD) techniques in an Oxford system attached with a Carl Zeiss AURIGA® field emission gun scanning electron microscopy (FEG SEM) workstation in order to investigate the presence of titanium nitride (TiN), silicon nitride (N_4Si_3) and aluminum oxide (Al_2O_3) phases. The EBSD pattern was optimized by using a 2×2 binning mode with a step size of 0.1 µm/s. Compression tests were conducted in an Instron 5982 equipped with BlueHill 3 analysis software and a 100 kN load cell under a loading rate of 0.5 mm/min.

3. Results and Discussion

3.1. Formation of a ceramic surface

In order to produce the gas required for surface hardening of the steel specimens, selected automotive shredder residue was incinerated at a temperature of 1200ºC. The organic components of the residue degraded as a function of time at temperature and carbon-saturated gas was produced as shown in Fig. 1. During this heat treatment, reactions occurred on the steel surface: aluminum melted and reacted with oxygen to form aluminum oxide; titanium oxide was converted to titanium nitride and silicon oxide was converted to silicon nitride. Steel is a catalyst for all these reactions and in the result, the reduction of titanium silicon oxides as well as the formation of titanium and silicon nitride occurred at temperatures lower than would be expected from standard thermodynamic considerations.

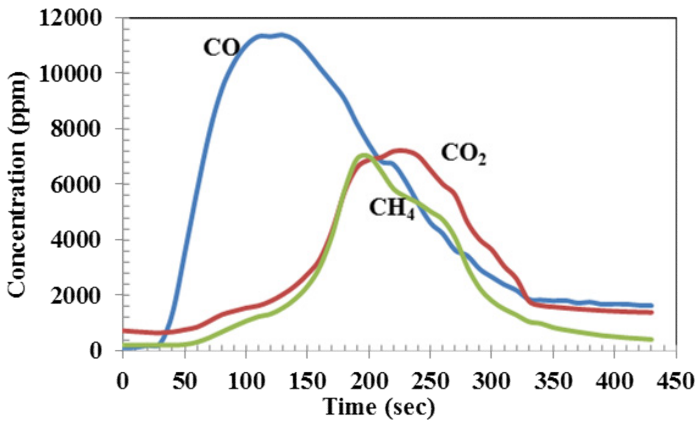

Fig. 1. Off gas analysing of heat-treated ASR at 1200ºC.

It is instructive to evaluate the formation of individual compounds.

3.2. Aluminum oxide

The selected automotive shredder residue (ASR) contained small amounts of aluminum, which is in the liquid state at 1200°C. Due to good chemical bonding and low wettability between aluminum and iron [21], the aluminum quickly covers the steel surface.

However, aluminum has a very strong chemical affinity for oxygen and tends to form aluminum oxide on the steel surface. Moreover, this oxidation reaction is exothermic, it will release energy and form local micro-reactors which results in the formation of aluminum oxide at neighboring sites. The XPS spectrum of Al2p in Fig. 2 shows the formation of this aluminum oxide on the steel surface following heat treatment at 1200ºC for 30 and 60 min.

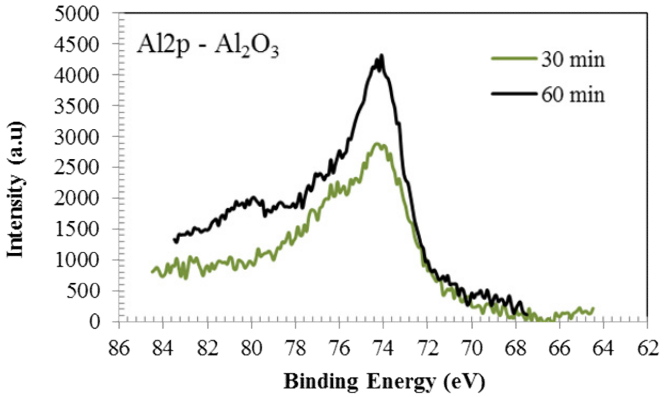

Fig. 2. XPS spectra of Al2p which shows the formation of Al_2O_3 on the surface of steel surface following reaction of 30 and 60 min. at 1200°C

3.3. Silicon Nitride

As well as carbon and nitrogen, ASR contains silicon in the form of S_iO_2, due to the presence of glass in the shredded waste mix. At 1200 ºC the reaction between the silicon oxide, reducing gases and carbon residue from the degradation of organic components of ASR will lead to the reduction of SiO2. During the process of SiO2 reduction, the presence of nitrogen from plastic leads to the formation of silicon nitride. The evidence for this is seen clearly in Fig. 3, which shows the XPS spectra of the Si2p results for the samples. Generally, the formation of silicon nitride needs a higher temperature and longer exposure time, but in this study iron acts as a catalyst to promote the formation of silicon nitride at a lower temperature and Ar acts as a carrier gas in these reactions.

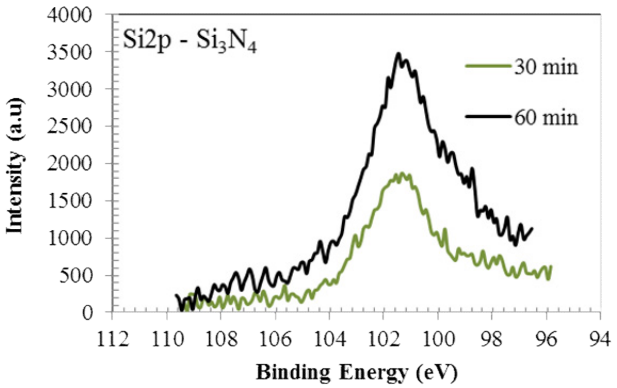

Fig. 3. XPS spectrum of Si2p, which shows the formation of Si_3N_4 on the steel surface following reaction of 30 and 60 min. at 1200°C

3.4. Titanium nitride

Another important component in the selected ASR is titanium oxide. Recent studies provided evidence that titanium oxide present in ASR can be reduced by carbon resulting from degraded organic components followed by nitridation to form TiN [22-24]. The XPS spectrum of Ti2p on the steel surface following reaction at 1200°C for 30 and 60 min. is shown in Fig. 4 and provides evidence of the formation of Ti-N bonds and transferring Ti-O bonds to Ti and then to Ti-N bonds.

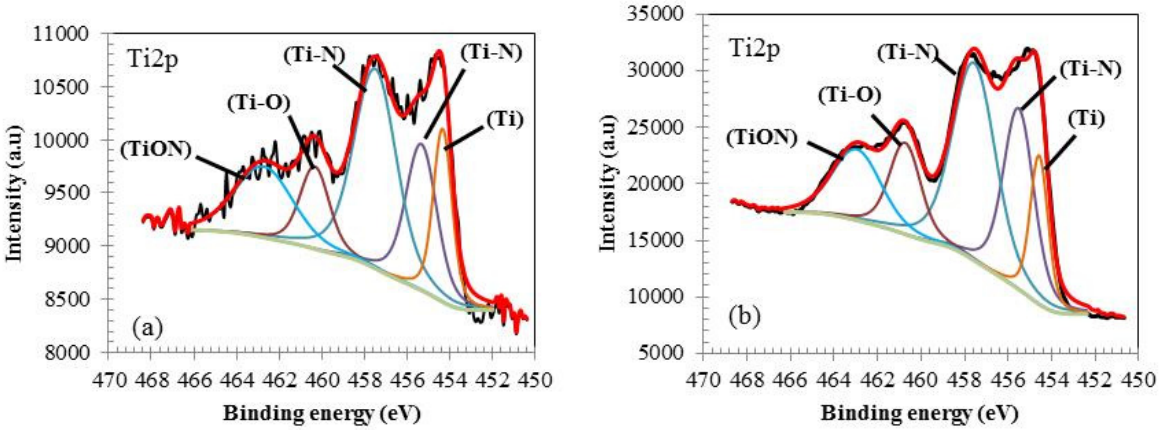

Fig. 4. XPS spectrum of a steel surface following reaction at 1200°C for a- 30 minutes and b- 60 minutes

3.5. Metallographic investigations

The cross-section of a sample heat treated at 1200 ºC for 30 minutes was investigated using optical microscopy and the EBSD techniques in order to reveal the morphology of the different ceramic phases on the sample surface. Fig. 5 shows that de-carburization occurred when the steel specimens were heated to at 1200°C and kept for 30 minutes at this temperature. Conversely, subjecting similar specimens to the identical heat treatment, but in the presence of ASR, a ceramic layer, containing aluminum oxide, Si_3N_4 and TiN) formed on the surface of the steel sample.

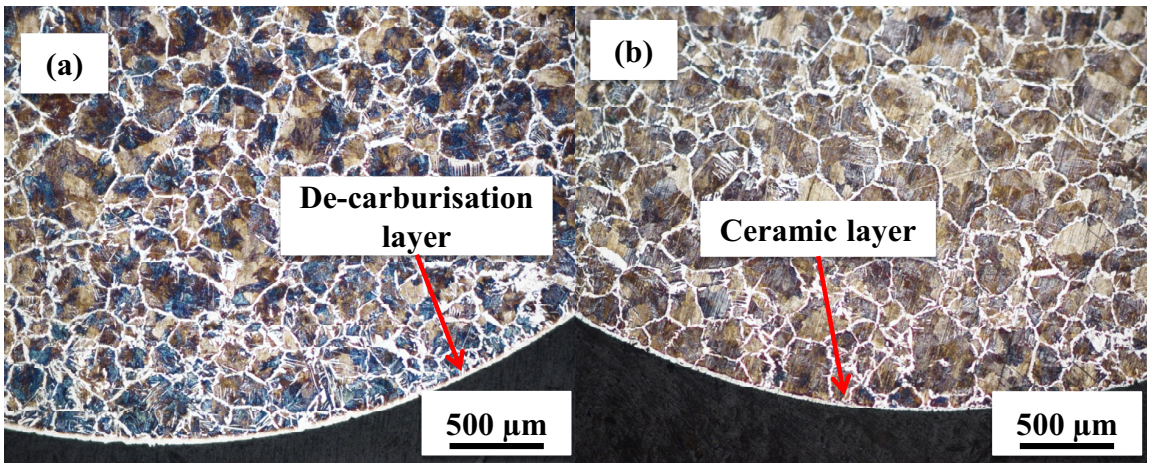

Fig. 5. Optical microstructure of steel after heat treatment at 1200ºC with and without ASR. a) heat treated without ASR, b) heat treated with ASR for 30min

TiN- and Al_2O_3-phases nucleated and grew uniformly while the Si_3N_4 -phase only nucleated and grew in some areas as shown in the EBSD image in Fig 6. These ceramic phases increase the hardness of the steel surface and since they are chemically bonded, they resist applied forces far better than physically bonded ceramic surfaces.

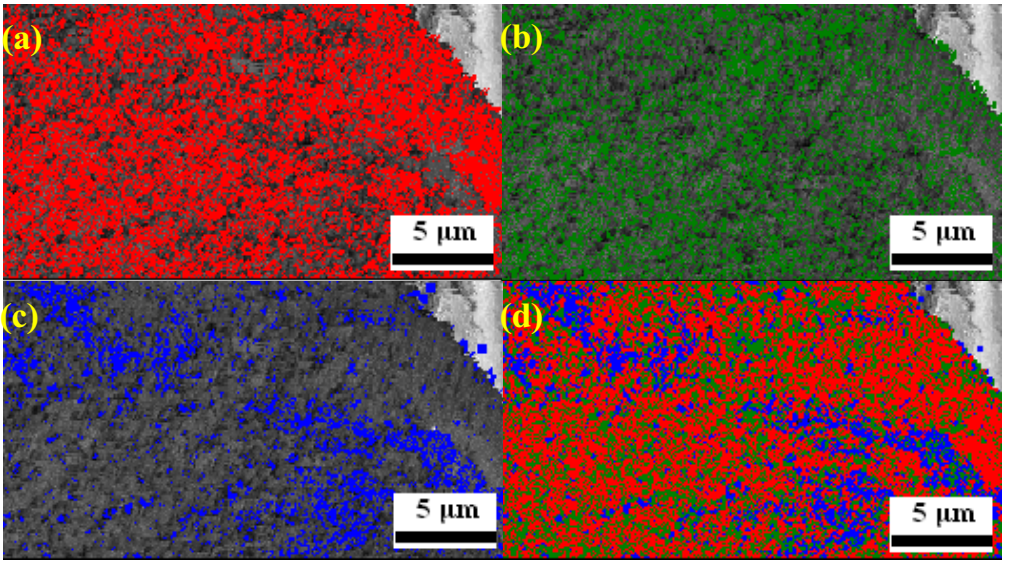

Fig. 6. EBSD Phase maps of a steel sample treated with ASR for 30min. at 1200°C. (a) TiN is shown as red (b) Al_2O_3 green (c) Si_3N_4 blue (d) combined EBSD phase map of all these elements. (Readers are referred to the web version of the article for colour interpretation.)

3.6. Mechanical properties

Following the heat treatments outlined above, the samples were subjected to compression tests to determine the effect of the resulting microstructures on the compression strength of the steel. Table 3 shows the compression strength of steel samples without any heat treatment (raw sample) and following heat treatment for 30 and 60 mins at 1200°C in the presence of ASR. The increase in compressive strength, shown in Table 3, takes into account the surface hardening in addition to other beneficial microstructural changes and should be a reasonable measure of efficiency in wear-resistant steel development.

Table 2: Compression strength after formation of ceramic surface

Sample	Compression strength (MPa)
Raw sample	885
1200°C – 30 min	940
1200°C – 60 min	950

4. Conclusions

In this research, the formation of a ceramic surface in situ on normal carbon steel by heat treating steel with automotive shredder residue has been investigated. XPS results indicate that after 30 minutes of heat treatment, aluminum oxide, and silicon and titanium nitride are formed on the steel's surface. Aluminum oxide formed on the steel surface in addition to silicon and titanium nitrides that nucleated and grew on the steel surface at lower temperatures than predicted by classical thermodynamics because of the catalytic properties of iron. From compression-strength measurements, it is clear that the formation of these in situ ceramic surfaces increases the

mechanical strength of normal carbon steel to exposure times of 30 minutes. By increasing the heat treatment time to 60 minutes, the increasing rate of compressive strength of the steel starts to decrease due to an increase in its grain size. However, due to an increase in the thickness of the ceramic surface, as well as the diffusion of carbon compression strength of steel has increased.

A logical implication of these preliminary investigations is that waste materials can be effectively used to enhanced the wear resistance of low cost steel while simultaneously reducing demands on expensive coatings technologies and in addition, to alleviate environmental damage caused by dumping automotive shredder residue in landfills.

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